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PROCESSING AND CHARACTERIZATION OF POLYCRYSTALLINE YAG (YTTRIUM ALUMINUM GARNET) CORE-CLAD FIBERS -POSTPRINT

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14. ABSTRACT
Polycrystalline YAG fiber has recently attracted considerable attention for the role it could play as a fiber-laser gain media. This primarily due to its large surface-to-volume ratio, high stimulated Brillouin scattering threshold, and its high thermal conductivity; all of which are superior to that of silica-glass fibers. As a consequence, techniques which enable the fabrication of poly- and single-crystalline YAG fibers have recently been the focus of a number of efforts. In this work we have endeavored to reduce the scattering loss of polycrystalline-YAG-core fibers while simultaneously demonstrating optical gain by enhancing our processing techniques using feedback from mechanical testing and through the development of a technique to encase doped YAG-core fibers with un-doped YAG claddings. To this end we have recently fabricated fibers with both core and claddings made up of polycrystalline YAG and subsequently confirmed that they indeed guide light. In this paper, the processes leading to the fabrication of these fibers will be discussed along with their characterization.

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Processing and characterization of polycrystalline YAG (Yttrium Aluminum Garnet) core-clad fibers

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ABSTRACT

Polycrystalline YAG fiber has recently attracted considerable attention for the role it could play as a fiber-laser gain media. This primarily due to its large surface-to-volume ratio, high stimulated Brillouin scattering threshold, and its high thermal conductivity; all of which are superior to that of silica-glass fibers. As a consequence, techniques which enable the fabrication of poly- and single-crystalline YAG fibers have recently been the focus of a number of efforts. In this work we have endeavored to reduce the scattering loss of polycrystalline-YAG-core fibers while simultaneously demonstrating optical gain by enhancing our processing techniques using feedback from mechanical testing and through the development of a technique to encase doped YAG-core fibers with un-doped YAG claddings. To this end we have recently fabricated fibers with both core and claddings made up of polycrystalline YAG and subsequently confirmed that they indeed guide light. In this paper, the processes leading to the fabrication of these fibers will be discussed along with their characterization.

Keywords: polycrystalline YAG, core-clad fiber, ceramic processing

1. INTRODUCTION

Due to the superior characteristics YAG possesses as a host gain media, which include its higher thermal conductivity and lower thermo-optic coefficients compared to glass, it has been considered an alternative host material for high-energy fiber lasers. Indeed, YAG-based fiber lasers would offer efficient operation at power levels beyond those achievable in current state-of-the-art silica-based fiber lasers if its losses can be minimized.[1,2] To address this researchers have investigated creating both single-crystal and polycrystalline YAG fibers. For example, Zhu et al. reported the preparation of single-crystal YAG fibers using laser heated pedestal growth (LHPG) which resulted in fiber diameters of 400 μm and optical losses around 1–2 dB/m in the 1–3 μm wavelength range.[3] Single-crystal YAG fibers with diameters of ~ 30 μm have even been reported.[4,5] These fibers were also prepared using LHPG and exhibited excellent optical qualities. Although single-crystal YAG fibers are now available, robust cladding processes have yet to be developed for them. While an number of cladding methods were investigated they still appear to be in their initial stages and lack the maturity required for the envisioned laser application.[4,6] Indeed, glass can be used as a cladding on single-crystal fibers but its thermal conductivity is about ten times lower than that of crystalline YAG. Therefore, it can only be used for characterization purposes as it negates the motivation to use YAG as a fiber media and is therefore not practical for actual applications.[2,7] Applying ceramic coatings to fully dense fiber is also problematic due to constrained sintering conditions which result in cracks forming in the cladding.[8] On the other hand, polycrystalline YAG fibers can be prepared with conventional ceramic processing, which involves a variety of processing steps including the formation of so-called "green fiber", the creation of binder-removed fiber, and finally fully dense fiber. In this study, green and binder-removed fibers were investigated for cladding studies using dip coating.

The processing steps used to make the polycrystalline YAG fibers studied in this work as well as the experimental characterization of the optical propagation losses in the resulting fibers have already been reported.[9-12] These losses were measured by injecting a 1480-nm fiber-coupled laser into these fibers which were cladded with 3 μm of Schott SF57 glass. Obviously the loss of these fibers, measured to be ≥ 50 1/m, needs to be significantly lowered to facilitate any practical application. Following this initial work it was discovered that these fibers all contained second-phase

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inclusions which likely lead to excess scattering loss. It was determined the cladding thickness was too thin to optimally confine the light within the fibers, and the refractive index difference between the YAG core and the glass cladding was too large at the characterization wavelength used. As a consequence of these findings, we have recently focused on removing the second-phase inclusions and on developing a process to apply an un-doped YAG cladding to a doped polycrystalline YAG fiber in order to optimize the refractive index difference between core and cladding. In this study we report on refined processing techniques able to routinely realize polycrystalline YAG fibers without second-phase inclusions (to date we have only made core-only fibers of this type). These findings are accompanied by the results of two types of older fibers which still possessed some degree of second-phase inclusions: YAG core-only and YAG core-clad geometries.

2. METHODOLOGY

2.1 Processing of green YAG fibers

As previously described (in Refs. 4–7) Yb-doped YAG powders were obtained from Nanocerox (Ann Arbor, MI). The as-received powder was subsequently ball-milled and classified to remove agglomerates and contamination from the milling media. Binder and plasticizer were mixed with the Yb-doped YAG powder and the mixture was extruded through a 50-μm custom-made nozzle at pressures 3000–5000 psi. The fibers were then dried at room temperature for ≥ 8 hours before any future process steps were taken.

2.2 Cladding and sintering of YAG green fibers

An un-doped YAG slurry was subsequently prepared (as described above) and the Yb-doped fibers were dip coated in this slurry multiple times to form the YAG cladding. After each dip coating, the coated fiber was dried at 120°C. Organics in the cladded green fiber were removed during slow ramping up to 600°C and the sintering was carried out under a variety of conditions.

2.3 Characterization of polycrystalline YAG fibers

To further investigate the composition of these fibers they were mounted, polished, and coated with carbon as a conductive layer for scanning electron microscopy (SEM, Quanta ESEM, FEI). Then the back-scattered electron mode of our SEM was used to analyze fibers for the presence of second-phase inclusions. In addition, focused ion beam (FIB, Nova, FEI) was used to prepare foils for energy dispersive x-ray spectroscopy (EDS) analysis via TEM (CM 200, Philips) to perform chemical analysis on the second-phase inclusions and YAG matrix. Optical scattering losses of these fibers were also measured using the approach detailed in Ref. 7.

3. RESULTS AND DISCUSSION

3.1 Second-phase observation and removal

While our earlier fibers guided light, the best loss coefficient was measured to be ~50 1/m. During the analysis of these fibers, however, it was found that they contained second-phase inclusions [as shown in Fig. 1 (a)]. This implies the loss of the fibers would improve if the second-phase inclusions could be removed.

The EDS spectra shown in Fig. 1(c) make it clear that the second-phase inclusions are Si rich and Al deficient. While it is still unclear what causes the formation of the second-phase inclusions, they are believed to increase the scattering losses of these fibers. After a number of experiments where the sintering conditions were changed, we found conditions which allowed us to create polycrystalline YAG fibers without inclusions; the microstructure of such a fiber is shown in Fig. 1(d). Scattering loss measurements for this fiber will be compared with those of earlier fibers in the near future.

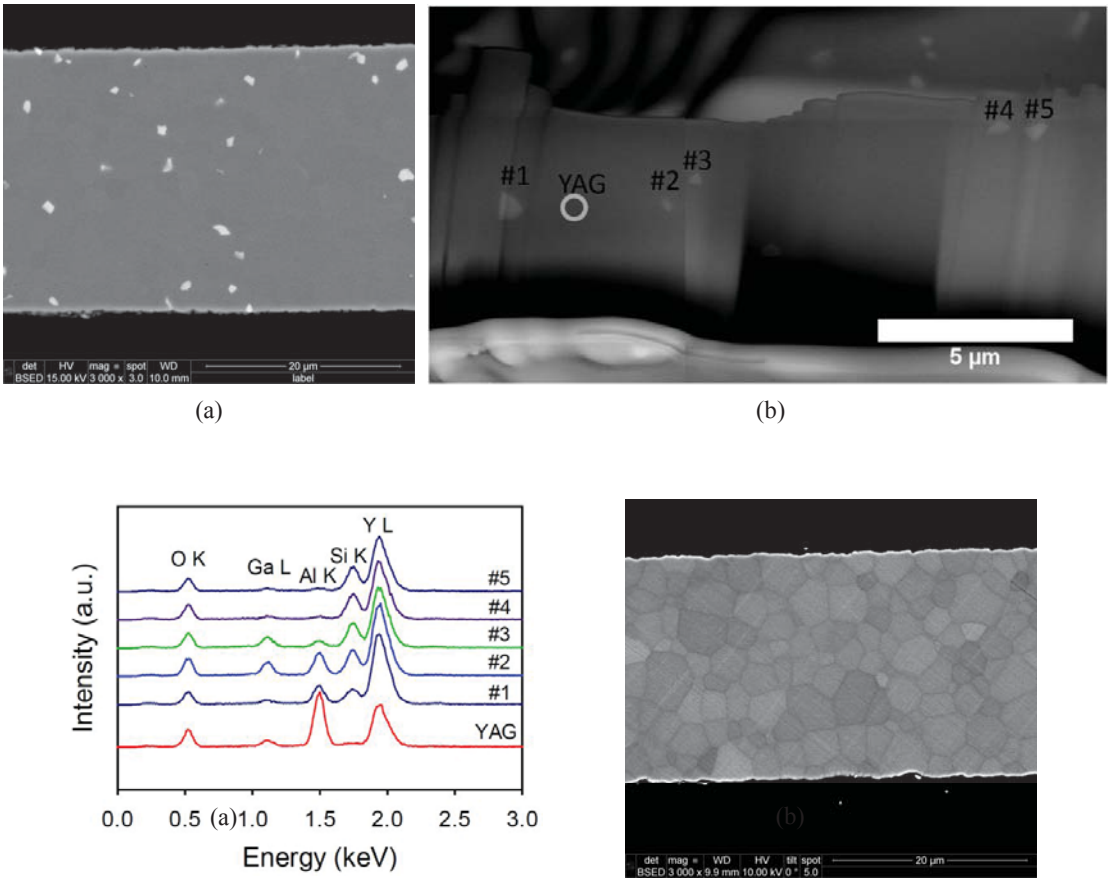


Fig. 1. SEM micrographs of cross-sectioned YAG fiber (a) and cross-sectioned foil (b) showing second-phase inclusions. EDS spectra (c) collected via TEM on matrix YAG and second-phase inclusions numbered in (b). SEM micrograph (d) of a polished section of YAG fiber without second-phase inclusions.

3.2 Applying un-doped polycrystalline YAG cladding to doped polycrystalline YAG fibers

Although the preparation of both single- and polycrystalline YAG fibers has been reported, robust cladding processes on those fibers have not yet been fully developed. Glass cladding was previously used on polycrystalline YAG fibers because it can be readily processed, however, surface-tension issues limited the thickness that could be reliably achieved. Moreover, the glass cladding was only applied for characterization purposes; it would limit the benefits of an all-YAG fiber in actual applications (e.g. its advantageous thermal properties). Since the fibers in this study were prepared with ceramic processing, they can easily be coated with an un-doped YAG slurry. Here dip coating was attempted on both

green and binder-removed fibers. However, because the organic system of the cladding slurry was similar to that of the extrusion mix, the binder in the green fiber dissolved when it came in contact with the slurry and further dip coating was not possible. As a result the achievable cladding thickness was limited. Nevertheless, dip coating the binder-removed fiber worked well and all cladded fibers in this study were prepared using this technique. Figure 2 shows the evolution of dip coating processes and the resulting fiber cross sections.

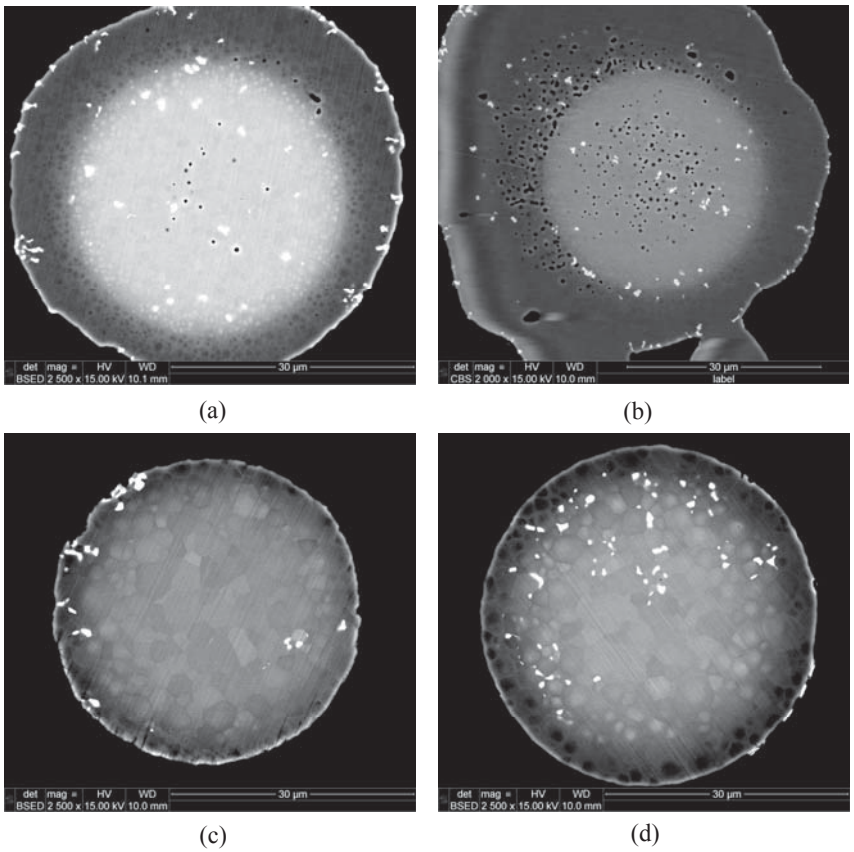


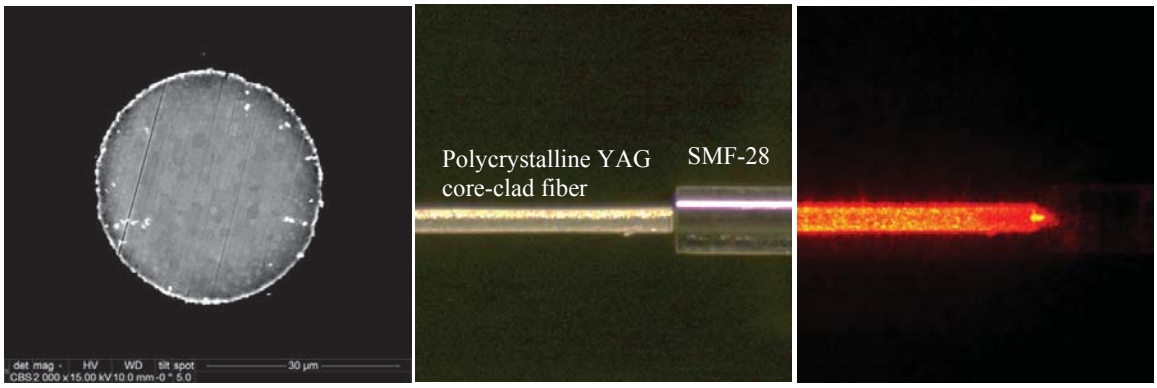
Fig.2. SEM micrographs of cross-sections of the polycrystalline YAG core-clad fibers: The evolution of our dip coating process can be seen by noting that the circularity of the cross sections and sintering temperatures improve from (a) to (d).

As shown in Fig. 2, Yb-doped cores look brighter than the surrounding cladding because the cores include a dopant with a higher atomic number. This generates brighter contrast under the back-scattered electron mode of the SEM. Initial characterizations were performed on the polycrystalline YAG core-clad fiber which was fabricated with the sintering temperature used for Fig. 2(c) followed by the dip coating method employed in Fig. 2(d). These characterizations are discussed in sections 3.3 and 3.4.

3.3 Preliminary characterization of the polycrystalline YAG core-clad fiber

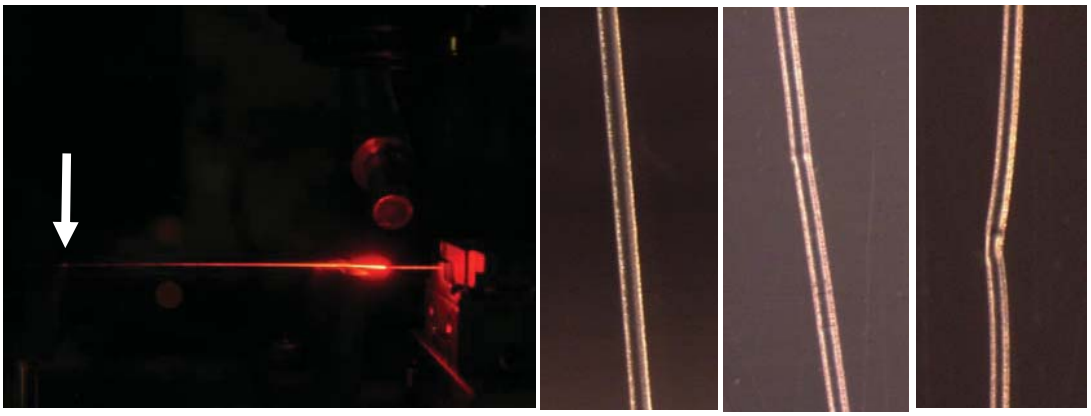
A roughly 2.5-inch long polycrystalline YAG core-clad fiber with a ~ 30-μm diameter whose microstructure is shown in Fig. 3 (a) was fabricated for characterization. The core region of this fiber appears to have a bright contrast and fills the majority of the cross-sectional area while the dark rim around the "circle" (barely visible in the figure) is the un-doped

YAG cladding. Ideally, the cladding should be thicker than that shown in Fig. 3(a). This has motivated us to explore other ceramic processes for the application of the cladding including co-extrusion and tape casting. We have also developed a new sintering process to prevent the formation of second-phase inclusions in the fiber (as discussed in section 3.1); however, the fiber in Fig. 3 was prepared with our original method and therefore still contains a small quantity of second-phase inclusions. Before performing optical loss measurements, the YAG fiber was butt-coupled to a SMF-28 delivery fiber where the alignment was optimized using a 632-nm fiber-coupled semiconductor laser. Figure 3(b) and (c) show the polycrystalline YAG fiber aligned to the SMF-28 fiber using the 632-nm laser. Figure 4 (a) shows the entire fiber guiding (and scattering) the 632-nm laser light. Figure 4(b) shows a well-prepared part of the fiber while the images in (b) and (c) reveal regions that include kinks. It was observed that the 632-nm laser light passes through the regions shown in (a) and (b) but that severe kinks, like the one shown in Fig. 4(d), ruin the wave-guiding abilities of these fibers. The kinks, such as the one highlighted by the arrow in Fig. 4(a), result in significant scattering as one would expect.



(a) (b) (c)

Fig. 3. SEM micrograph of the cross-section of polycrystalline YAG core-clad fiber (a) and alignment of the YAG fiber with SMF-28 delivery fiber (b) and (c).



(a) (b) (c) (d)

Fig.4. 2.5-inch polycrystalline YAG core-clad fiber scattering and guiding 632-nm laser light (a). Optical micrographs of a well prepared region of this fiber (b) and regions exhibiting kinks (c) and (d). The laser light stops being guided in (a) at the location identified by the arrow where the kink shown in (d) exists.

3.4 Scattering loss of polycrystalline YAG core-clad fiber and Yb excitation

Spatially resolved optical scattering collected from the polycrystalline YAG core-clad fiber is shown in Figs. 5–7 and was measured using the same general setup as that described in Ref. 7. Nevertheless, a subtle modification to our system was made to facilitate these measurements because these fibers were doped with Yb. Specifically, in this work a SMF-28 fiber-based wavelength-division multiplexing (WDM) coupler was used to spectrally resolve our measurements. While the WDM coupler was used for its expediency, it required our scanning "probe" fiber be switched from a multi-mode to a single-mode fiber. This greatly decreased the solid angle over which scattered light is collected and, while this could be used to improve our spatial resolution, it also greatly reduced the power levels incident in our integrating sphere. As a consequence, the signal-to-noise ratio for the data presented below is not as high as that obtained in our previous measurements (Ref. 7).

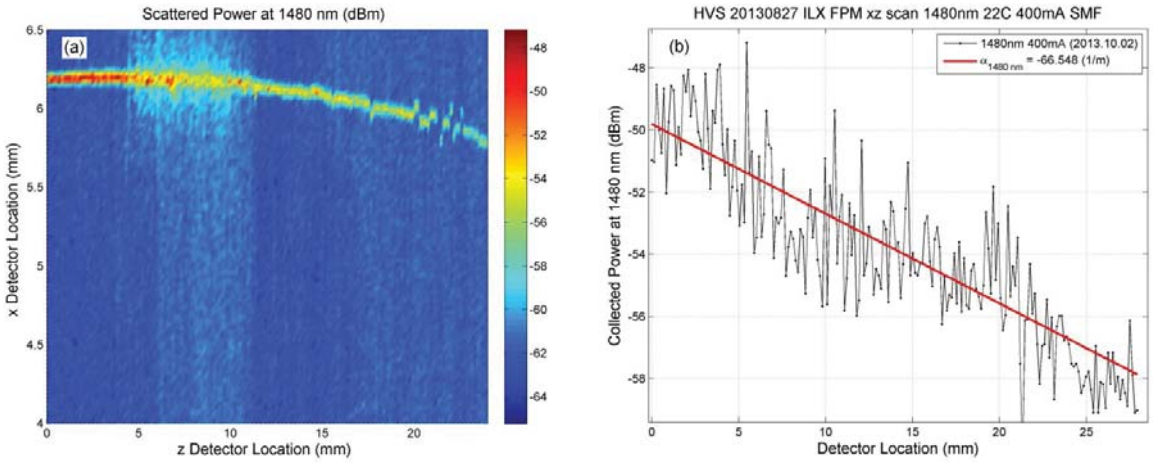


Fig.5. Optical power detected in an integrating sphere using a cleaved SFM-28 fiber as a probe tip and a 1480-nm pump laser.

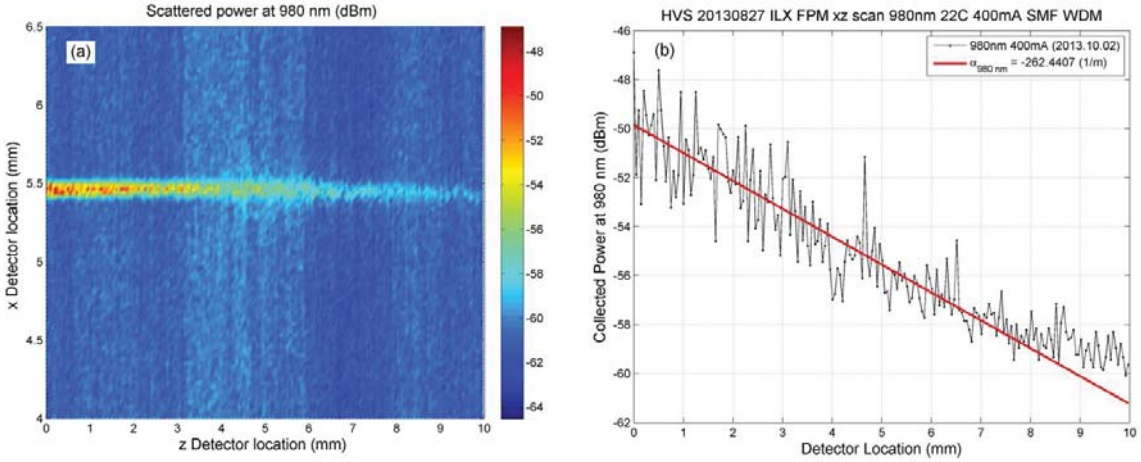


Fig. 6. Optical power detected in an integrating sphere using a cleaved SMF-28 fiber as a probe tip in conjunction with a 980/1030-nm WDM coupler to provide spectral resolution. In this case the 980-nm output port was used. (The fiber tested here is the same as that investigated in Fig. 5 however the fiber was re-aligned which shifted its location from ~ 6.2 to 5.5 mm)

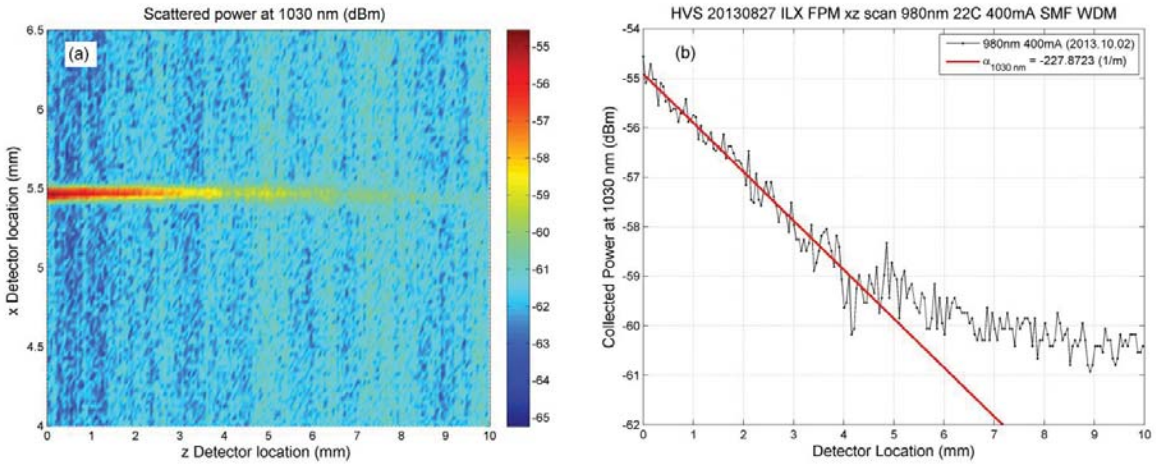


Fig. 7. Optical power detected in an integrating sphere using a cleaved SMF-28 fiber as a probe tip in conjunction with a 980/1030-nm WDM coupler to provide spectral resolution. In this case the 1030-nm output port was used.

While the optical losses measured for this initial fiber were significantly larger than those found in previous work (50 1/m) we believe this to be attributable to our initial YAG cladding dimensions which were far too thin to ensure a negligible optical field at the cladding–air interface. Still, despite the high losses measured at 1480 nm, we measured a considerably higher loss at 980 nm. While scattering-induced losses should increase at lower wavelengths the discrepancy between the losses at these two wavelengths is far more extreme that than observed in previous work.[7] Clearly the large increase in loss at 980 nm is due to the Yb doping of the core of these fibers which absorbs a significant amount of the 980-nm pump light. Figure 7 shows the combination of spontaneous emission (SE) and scattered amplified spontaneous emission (ASE) which is subsequently scattered out of this fiber in the ~1030–1050 nm regime identifying that part of this fiber is inverted.

4. CONCLUSIONS

Polycrystalline YAG core-clad fibers were fabricated and found to guide light at wavelengths separated by 500 nm. The scattering loss coefficient of the fiber was measured to be below 70 1/m at 1480 nm and while this value is higher than that of our previous fibers (whose loss coefficient was ~40 1/m) these fibers were doped. The excess loss has also been attributed to excess scattering at the cladding-air interface and the lingering presence of second-phase inclusions which we only recently managed to suppress. New fibers with thicker claddings and a lack of second-phase inclusions will be optically tested in the near future.

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